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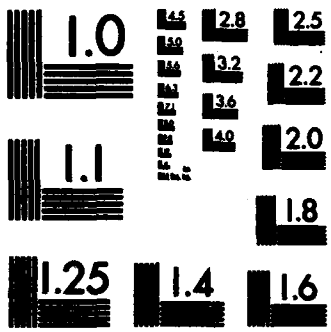


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# FLAMMABILITY CHARACTERISTICS OF SOME EPOXY RESINS AND COMPOSITES

DOMENIC P. MACAIONE, RICHARD P. DOWLING, II,
and PAUL P. BERGQUIST
POLYMER RESEARCH DIVISION

September 1983



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## ABSTRACT

The flammability characteristics of a number of epoxy resin formulations and glass fiber-reinforced epoxy resin composites have been evaluated by thermal analysis, limiting oxygen index/temperature index, flash ignition, and smoke density measurement techniques. Results have indicated that appropriate flame-retardant additives or halogenated monomers should be incorporated into the matrix resin to increase materiel survivability and reduce resin combustibility.

CONTENTS

# INTRODUCTION

Organic polymers which are of present or potential interest as components of military hardware must be evaluated in terms of their flammability characteristics in order to gain insight into their ability to resist a flame threat and to reduce the hazards to personnel while increasing the survivability of equipment. One widely used class of organic polymers in such applications is epoxy resins. These materials are either being used, or considered for use, in aircraft, tank and automotive, electronic, bridge and building, and weapon/munitions applications.

Hundreds of epoxy resins have been prepared on an experimental basis, including polymers with aliphatic, alicyclic, and aromatic backbones. There seem to be no reliable figures estimating the usage and applications of epoxy resins. Of the many informed guesses the following division may be used as a guide:

| Applications | Percent of Production |
|---|---|
| Coatings | 50 |
| Composite Matricies | 25 |
| Casting Resins | 10 |
| Adhesives | 5 |
| Other Applications | 10 |

The investigation reported herein is primarily concerned with epoxy materials as casting resins and/or composite matricies since relatively large concentrations of resin, per unit area of material, exist in these applications, and would, therefore, present the greatest flammability hazard.

# EXPERIMENTAL

## Thermogravimetric Analysis (TGA)

A thermogravimetric analysis system consisting of a DuPont 990 Thermal Analyzer and a 951 TGA module was employed to determine weight loss (a) as a function of temperature, and (b) isothermally, as a function of time; in flowing air at a present flow rate of 50 cc/minute for each material. The resultant data is an indication of thermal stability of the material. In general, materials which are thermally stable are less flammable than those which are thermally labile since the concentration of combustible low molecular weight fragments is reduced for a given temperature up to the point where major decomposition occurs.

## Oxygen Index/Temperature Index Analysis (OI/TI)

As a measure of the susceptibility to ignition, the limiting oxygen index value was determined with a Stanton-Redcroft FTA apparatus for each sample and each material was evaluated at elevated temperatures with a Stanton-Redcroft HFTA apparatus to determine the temperature index profile. Essentially, this consists of a measurement of the minimum oxygen concentration of a flowing oxygen/nitrogen mixture which is required to support equilibrium burning of a vertically-oriented 1/4" x 1/2" x 3" sample of the material under test at temperatures from ambient to 300°C.

## Smoke Density Measurement

The smoke density value for each sample material was determined in an Aminco-NBS Smoke Chamber. This unit incorporates a vertically-oriented photometer in a chamber volume of 18 cubic feet to evaluate the density of smoke generated by the combustion of a 3" x 3" test sample in either smoldering or flaming mode. In the former condition, the sample is subjected to the thermal energy of an electric furnace such that the face of the sample receives 2.5 watt/cm$^2$. In this condition, the surface temperature is on the order of 350°C. In the latter condition, the thermal energy of the electric furnace is augmented by six small flame jets from a propane-air burner. The smoke density value is determined as the decrease in light transmission measured by the photometer. Values of "maximum optical density" ($D_m$) and/or "specific optical density" ($D_s$) are quoted, as appropriate. Larger values indicate that more smoke is produced by the sample material.

## Flash Ignition Temperature

Samples were examined for flash ignition temperature by placement of a pilot flame above the exit port of an autoignition apparatus consisting of a 3-inch-diameter furnace tube which is electrically heated and through which air is passed at a present flow rate. The sample is held in a ceramic cup within the furnace, and the temperature within the heating coil, air stream, sample cup, and sample are monitored with thermocouples. The flash ignition temperature is that sample temperature at which sufficient combustible vapor is mixed with air and passes upward to the pilot flame to cause flashback and ignition of the main sample.

## Resin and Composite Formulations

### A. Resins

Epoxy resin samples were prepared (see Table 1) based upon Epon 828 and various curing agents in order that the flammability behavior of typical epoxy resin systems could be determined. In addition, three resin systems (see Table 2) employed as composite matricies were evaluated in the neat state to determine the baseline flammability response for the matrix.

Table 1. EPOXY RESIN SYSTEMS

| Resin | Curing Agent | Resin/Curing Agent | Cure Cycle |
|---|---|---|---|
| Epon 828 | Tonox 60/40 | 100/20 | 2 h/80°F<br>2 h/150°F |
| Epon 828 | Versamid 140 | 100/30 | Ambient Temperature |
| Epon 828 | Triamine 403 | 100/60 | 3 h/100°C |
| Epon 828 | Nadic Methyl Anhydride (NMA) | 100/95 | 2 h/93°F<br>4 h/150°F |

Table 2. MODIFIED EPOXY RESIN SYSTEMS

| System No. | Resin | Curing Agent | Cure Cycle |
|---|---|---|---|
| 1009* | Epon 828/DEN438‡ | $BF_3$-Monoethyl Amine (MEA) | 45 min/160°C<br>0-4 h/177°C |
| SP-250* | Epon 828/ECN** | Dicy/Monuron | 2 h/260°F‖ |
| RAC-7250† | TGMDA§/ECN | Dicy/Diuron | 2 h/260°F‖ |

*Minnesota Mining & Mfg. Co.
†Reliable Mfg. Co.
‡Dow Epoxy Novalac
**Epoxy Cresol Novalac
§Tetraglycidylmethylene Dianiline
‖Standard cure cycle is complex; essential portion is two hours at 260°F ± 5°F.

### B. Fiber-Reinforced Composites

Samples of fiber-reinforced epoxy resin composite systems were examined (see Table 3), as received, to evaluate if possible, any effect of the reinforcement on the flammability behavior of the composites. The samples consisted of 14-ply laminates of epoxy resin and glass fiber in the average ratio 30% resin to 70% glass fiber.

Table 3. FIBER-REINFORCED COMPOSITE SYSTEMS

| Resin | Fiber | Fiber Direction |
|---|---|---|
| 1009 | Glass | Unidirectional |
| SP-250 | S2-Glass | Unidirectional |
| SP-250 | E-Glass | Unidirectional |
| SP-250 | S2-Glass | Cross-Ply |
| SP-250 | E-Glass | Cross-Ply |
| RAC-7250 | S2-Glass | Unidirectional |
| RAC-7250 | E-Glass | Unidirectional |
| RAC-7250 | S2-Glass | Cross-Ply |
| RAC-7250 | E-Glass | Cross-Ply |

## RESULTS

### Thermogravimetric Analysis (TGA)

The results of thermogravimetric analysis experiments to determine the resin decomposition temperature (RDT) and isothermal decomposition rate of the cured matrix resins are shown in Table 4.

Table 4. DECOMPOSTION TEMPERATURES AND RATE FOR CURED EPOXY RESINS

| Resin | Curing Agent | RDT (°C) | Decomp. Rate* (mg/h at 250°C) $\times 10^{-2}$ |
|---|---|---|---|
| Epon 828 | Tonox 60/40 | 390 | 6 |
| Epon 828 | Versamid 140 | 373 | 5 |
| Epon 828 | Triamine 403 | 378 | 11 |
| Epon 828 | NMA | 405 | 6 |
| 1009 | $BF_3$-MEA | 412 | 5 |
| SP-250 | Dicy/Monuron | 300 | 4 |
| RAC-7250 | Dicy/Diuron | 305 | 3 |

*Conducted in flowing air atmosphere; flow rate = 50°C/min.
Calculated for approximately 30% loss from original mass of sample.

### Oxygen Index/Temperature Index Analysis (OI/TI)

The results of experimental measurements made to determine the oxygen index of the cured epoxy resins are shown in Table 5 and the results of similar experiments on the 1009 system to determine the effect of curing conditions upon the oxygen index are presented in Table 6.

Table 5. OXYGEN INDEX VALUES OF CURED EPOXY RESINS

| Resin | Curing Agent | Oxygen Index |
|---|---|---|
| Epon 828 | Tonox 60/40 | 20 |
| Epon 828 | Versamid 140 | 19 |
| Epon 828 | Triamine 403 | 18 |
| Epon 828 | NMA | 19 |
| 1009 | $BF_3$-MEA | 18 |
| SP-250 | Dicy/Monuron | 25 |
| RAC-7250 | Dicy/Diuron | 29 |

Table 6. EFFECT OF CURE CONDITIONS ON THE OXYGEN INDEX OF CURED 1009 RESIN

| Sample | Cure Condition (min/°C) | Oxygen Index |
|---|---|---|
| A | 45/160 | 21.7 |
| B | 45/160<br>60/177 | 21.3 |
| C | 45/160<br>120/177 | 21.9 |
| D | 45/160<br>180/177 | 21.7 |
| E | 45/160<br>240/177 | 21.5 |

The results of experimental measurement of the oxygen index as a function of temperature to determine the temperature index profile are presented in Table 7 and graphically in Figure 1.

Table 7. TEMPERATURE DEPENDENCE OF THE OXYGEN INDEX FOR CURED EPOXY RESINS

| | | Oxygen Index (°C) | | | |
|---|---|---|---|---|---|
| Resin | Curing Agent | 25 | 100 | 200 | 300 |
| Epon 828 | Tonox 60/40 | 20.0 | 19.3 | 16.6 | 16.1 |
| Epon 828 | Versamid 140 | 19.0 | 18.1 | 17.0 | 15.4 |
| Epon 828 | Triamine 403 | 18.2 | 17.7 | 16.6 | 15.8 |
| Epon 828 | NMA | 18.6 | 17.4 | 16.3 | 15.5 |
| 1009 | $BF_3$-MEA | 18.3 | 17.3 | 16.7 | 15.5 |
| SP-250 | Dicy/Monuron | 24.8 | 25.3 | 24.2 | 19.0 |
| RAC-7250 | Dicy/Diuron | 29.0 | 36.5 | 37.0 | 16.0 |

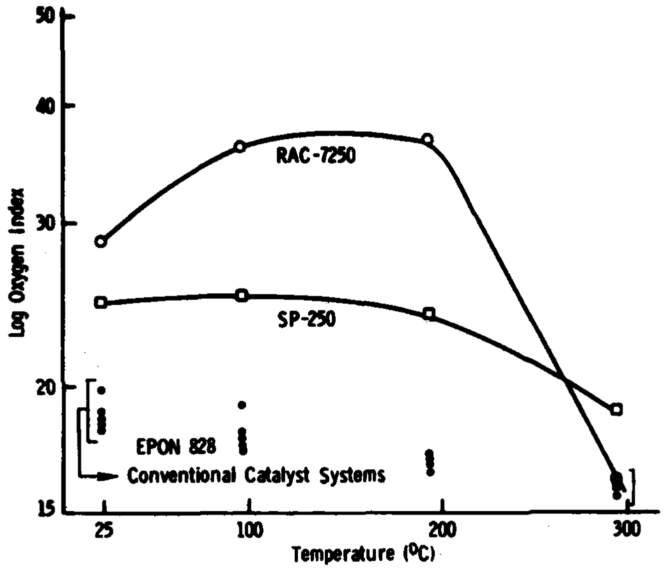



Figure 1. Epoxy resin systems.

A similar set of experiments was conducted with the SP-250, RAC-7250, and the 1009-26 Glass-Fiber Reinforced Composite Systems to evaluate the temperature index profile of these composites. The results are shown in Table 8 and graphically in Figure 2.

Table 8. TEMPERATURE DEPENDENCE OF OXYGEN INDEX FOR GLASS-FIBER REINFORCED COMPOSITES

| Composite System | Oxygen Index (°C) | | | |
|---|---|---|---|---|
| | 25 | 100 | 200 | 300 |
| RAC-7250-S2-C | 50 | 45 | 36 | 31 |
| RAC-7250-E-C | 56 | 43 | 28 | 25 |
| RAC-7250-S2-U | 55 | 52 | 38 | 25 |
| RAC-7250-E-U | 61 | 48 | 38 | 24 |
| SP-250-S2-C | 42 | 39 | 33 | 24 |
| SP-250-E-C | 43 | 41 | 34 | 32 |
| SP-250-S2-U | 55 | 47 | 42 | 38 |
| SP-250-E-U | 42 | 34 | 26 | 25 |
| 1009-26 | 41 | 39 | 26 | 23 |

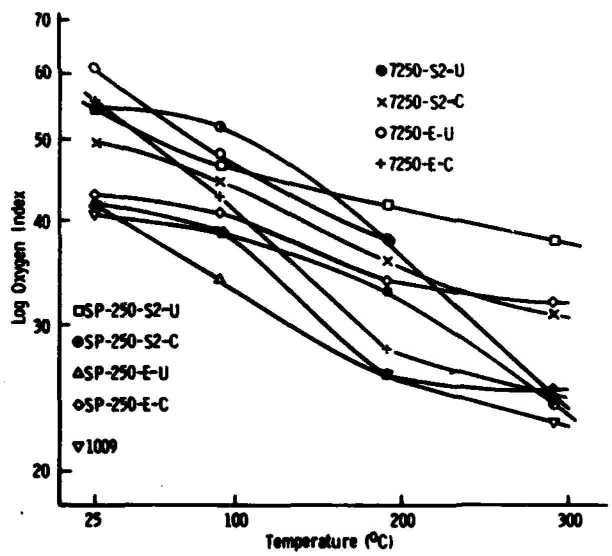



Figure 2. Epoxy-glass composites.

## Smoke Density Measurements (SD)

To evaluate the smoke generation capability of the epoxy resin and glass-fiber reinforced epoxy composite samples, a series of experiments were conducted with three-inch-square specimens in the NBS Smoke Density Chamber. The results obtained are shown in Tables 9 and 10.

Table 9. SMOKE DENSITY MEASUREMENT OF EPOXY RESINS

| Resin | Curing Agent | $D_m$ (corr) | Time to $D_s$ = 16 (sec) |
|---|---|---|---|
| Epon 828 | Tonox 60/40 | 823*<br>853† | 221*<br>84† |
| Epon 828 | Versamid 140 | 871*<br>883† | 187*<br>64† |
| Epon 828 | Triamine 403 | 874*<br>872† | 136*<br>55† |
| Epon 828 | NMA | 835*<br>872† | 156*<br>67† |
| 1009 | $BF_3$-MEA | 362*<br>416† | 121*<br>55† |
| SP-250 | Dicy/Monuron | Reliable data could not be obtained due to rapid pressure changes which ruptured the safety panel. | |
| RAC-7250 | Dicy/Diuron | | |

*Smoldering mode test
†Flaming mode test
$D_m$ = 264 is equivalent to 1% light transmission.
$D_s$ = 16 is time required to reach 75% light transmission.

Table 10. SMOKE DENSITY MEASUREMENT OF GLASS-FIBER REINFORCED EPOXY RESIN COMPOSITES

| Composite | $D_m$ (corr) | Time to $D_s$ = 16 (sec) |
|---|---|---|
| RAC-7250-S2-C | 313*<br>275† | 69*<br>83† |
| RAC-7250-E-C | 315*<br>250† | 70*<br>85† |
| RAC-7250-S2-U | 300*<br>275† | 60*<br>98† |
| RAC-7250-E-U | 458*<br>274† | 45*<br>67† |
| SP-250-S2-C | 288*<br>310† | 117*<br>102† |
| SP-250-E-C | 376*<br>307† | 135*<br>99† |
| SP-250-S2-U | 266*<br>330† | 101*<br>96† |
| SP-250-E-U | 247*<br>313† | 133*<br>90† |
| 1009-26 | 141*<br>121† | 282*<br>117† |

*Smoldering mode test
†Flaming mode test

### Flash Ignition Temperature (FIT)

The flash ignition temperature of epoxy resin and composite samples was measured in the apparatus described earlier. Air flow through the furnace was set at 9 liter/minute and the weight of the sample was chosen so that ignition, if it occurred, could be readily observed. Resin samples were 2-3 grams per experiment and composite samples were on the order of 9-10 grams. The results of these experiments are shown in Table 11.

Table 11. FLASH IGNITION TEMPERATURE OF EPOXY RESINS AND COMPOSITES

| Resin/Composite | Curing Agent | FIT (°C) |
|---|---|---|
| Epon 828 | Tonox 60/40 | 367 |
| Epon 828 | Versamid 140 | 356 |
| Epon 828 | Triamine 403 | 355 |
| Epon 828 | NMA | 373 |
| 1009 | $BF_3$-MEA | 394 |
| SP-250 | Dicy/Monuron | 364 |
| RAC-7250 | Dicy/Diuron | 328 |
| 1009-26 | - | 410 |
| SP-250-E | - | 373 |
| RAC-7250-S2 | - | 352 |

## DISCUSSION

Results of the thermogravimetric analysis experiments indicate that major decomposition of the epoxy resins initiates over a range of nearly 100 centigrade degrees and that the rate of decomposition spans nearly one order of magnitude. It is interesting to note that the epoxy resins with the lowest decomposition temperature have the slowest rate of decomposition. In terms of their limiting index oxygen values, the Epon 828 systems react similarly, even to the 1009 epoxy which contains Epon 828; however, SP-250 and RAC-7250 exhibit higher LOI values indicating that these two resins are more difficult to ignite. This would seem to agree with the findings that these resins have low decomposition rates in the isothermal TGA experiments.

Experimental results on the effect of cure conditions versus LOI would indicate that no essential change in the oxygen index is observed once the system has been through the initial cure portion of the cure cycle so that the presence or absence of post-curing makes no contribution to the flammability characteristics of the material.

By evaluation of the temperature dependence of the oxygen index one goes beyond the aspect of ignition properties of a particular material and considers the behavior of the material once ignited. The temperature index profiles of all but the SP-250 and RAC-7250 systems are essentially similar. These two systems are more difficult to ignite (higher LOI values) and show some increase in the oxygen index as a function of temperature but their final values at 300°C are approximately those of Epon 828 resin systems. The increase in the oxygen index of these resin systems in the 100°C-200°C range could be caused by the release of some compound in the vapor phase which acts as a flame retardant until it is lost or destroyed above 200°C and the temperature index turns downward. The temperature at which this inflection occurs is important because it indicates the point at which the material becomes more flammable and the hazard rating increases. The shape of the data curve is, obviously, also important since a very sharp decline in the oxygen index would indicate that the material would tend to combust rapidly and perhaps with considerable force.

When the basic resin material becomes the matrix of a composite the reinforcing material acts a a diluent for the combustible resin, thus, the observed values of the limiting oxygen index tend to increase and the temperature index profile is shifted upward. (This assumes, of course, that the reinforcing material of which the composite is made is not, itself, combustible.) The composite is apparently less flammable than the pure resin, but, it would appear more likely that these observations come about because there is less resin present in the composite, per unit weight, to act as a fuel source. Overall, the experiments conducted with the glass-fiber reinforced epoxy resins do not seem to indicate any change in flammability behavior that can readily be associated with changes in the matrix resin.

The experimental results of the smoke density measurements clearly indicate that epoxy resins are among the greatest smoke producers that one encounters. Smoke density maxima in the range of 800 to 900 units translates to light transmission values on the order of $1 \times 10^{-4}$ percent which is strictly academic since in any realistic situation, the limits of visibility are exceeded long before these values are reached. When the basic resin is incorporated into a composite the maximum smoke density value is reduced undoubtedly due to the diluent effect. Most of the composite samples still exceed a smoke density which translates to less than one percent ambient light transmission.

The results of the flash ignition experiments indicate that epoxy resin would be expected to ignite well within the range of temperatures that the material might experience in proximity to a fire, thus, adding to the total fire load and, as indicated above, adding in a significant way to the total smoke load experienced.

## CONCLUSIONS

The experimental observations presented herein indicate some of the potential fire hazards associated with epoxy resins and fiber-reinforced composites which employ an epoxy matrix. This effort clearly indicates that, as a component of military hardware, epoxy resins must be formulated to possess greater fire resistance which will increase materiel survivability and personnel safety.

To some degree, this might be accomplished with the addition of flame-retardant additives to the resin formulation, or by the copolymerization of halogenated species into the backbone of the matrix resin. This would be expected to increase the smoke generation problem but these resins already possess large baseline values of specific optical density, and the increase engendered by the presence of the flame retardant should have little overall effect on the value of the maximum optical density observed in the smoke chamber experiments.

As is always true, in cases of resin formula modification by addition of fillers or flame retardants, changes in resin formulation must be balanced against loss of beneficial physical properties.

## ACKNOWLEDGMENT

The authors wish to thank Mr. Samuel Hargis and Mr. Clifford Jacobs, members of the Northeastern University Cooperative Education Program, for their assistance with some of this work.

## APPENDIX A.

During the course of this work, and a number of isolated experiments not reported elsewhere, data on the flammability of epoxy resins and/or composite materials were obtained. That information is presented herein:

### I. Temperature Index Profiles

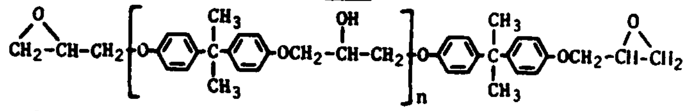
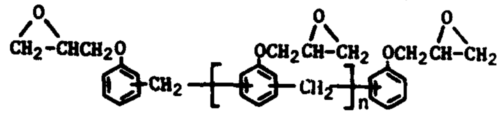
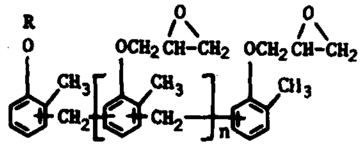
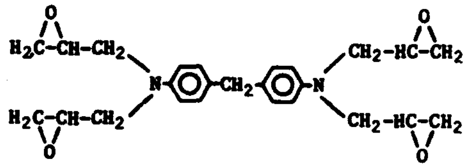
| Experimental Composite | Temperature (°C) | Oxygen Index |
|---|---|---|
| a) Kevlar 49/APCO 2434 | 25 | 26 |
| | 100 | 25 |
| | 200 | 24 |
| | 300 | 19 |
| b) Graphite Fiber/5208 Epoxy | 25 | 25 |
| | 100 | 23 |
| | 200 | 19 |
| | 300 | 18 |
| c) Kynol/828 Epoxy | 25 | 22 |
| | 100 | 21 |
| | 200 | 18 |
| | 300 | 16-17 |
| d) Kevlar/XD7818-T403 Epoxy | 25 | 25 |
| | 100 | 21 |
| | 200 | 19 |
| | 300 | 18 |

### II. Oxygen Index of Reinforcing Materials

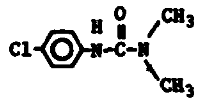
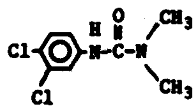
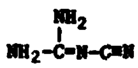
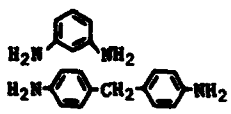
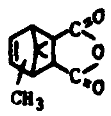
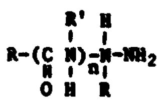
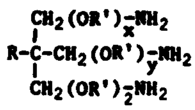
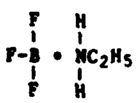
| Material | Oxygen Index |
|---|---|
| Kevlar 49 | 29 |
| Kevlar 29 | 31 |
| Kynol | 24-25 |

# APPENDIX B. EPOXY RESINS AND CURING AGENTS.

## I. Epoxy Resins:

| Trade Name | Number | Type |
|---|---|---|
| 1) Epon | 828 |  |
| 2) DEN | 438 |  |
| 3) ECN | |  |
| 4) TGMDA | |  |

## II. Curing Agents:

| | |
|---|---|
| 1) Monuron |  |
| 2) Diuron |  |
| 3) Dicyandiamide (DICY) |  |
| 4) Tonox 60/40<br>60% m-phenylenediamine<br>40% 4,4'-diaminodiphenylmethane |  |
| 5) NMA (nadic methyl anhydride) |  |
| 6) Versamid 140 |  |
| 7) Triamine 403 |  |
| 8) $BF_3$-MEA |  |

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